A2LA Assessor Environmental Method Checklist

Inductively Coupled Plasma - Atomic Emission Spectromet

Item	Section 1 - Personnel	Reference	Yes-No or NA	
1.1	Does the analyst(s) interviewed meet the job description position requirements, training and qualifications for performing the test? Supervisor:_____ Technician:_____	(G25)6.1		

Item	Section 2 - Equipment & Facilities	Reference	Yes-No or NA	
2.1	Is an ICP emission spectrometer equipped with computer controlled emission spectrometer, background correction, radio frequency generator compliant with FCC regulations, Argon gas supply, sample delivery pump and an optional mass flow controller?	(ORDM)200.7,6.1 (5/94)		
2.2	Is a temperature adjustable hot plate or block digestor used for sample preparation?	(ORDM)200.7,6.3 (5/94)		

Item	Section 3 - Method	Reference	Yes-No or NA	
3.1	Is glassware cleaned by washing with a detergent solution, rinsing with tap water, soaking for 4 hours or more in 20% (v/v) nitric acid or dilute mixture of nitric and hydrochloric acid (1+2+9), rinsing with reagent water and storing clean?	(ORDM)200.7,6.10 (5/94)		
3.2	Are all reagents high purity, conforming to ACS specifications, and all acids ultra high purity grade or equivalent. Are they analyzed for contamination if purity is in question?	(ORDM)200.7,7.1 (5/94)		
3.3	Is ASTM Type I water used for reagent water?	(ORDM)200.7,7.4 (5/94)		
3.4	Is the instrument allowed to thermally stabilize for at least 30 - 60 minutes before calibration and analyses?	(ORDM)200.7,11.4.3 (5/94)		

3.5	Are mixed calibration standard solutions prepared for total recoverable digested samples using stock solutions of individual elements in 500 mL volumetric flask with 20 mL (1+1) nitric acid and 20 mL (1+1) hydrochloric acid?	(ORDM)200.7,7.9 (5/94)		
3.6	Are the calibration standards prepared from non-certified primary standards initially verified using a certified reference solution?	(ORDM)200.7,7.9 (5/94)		
3.7	Are four blanks (calibration blank, laboratory reagent blank, laboratory fortified blank, and a rinse blank) analyzed as part of the method?	(ORDM)200.7,7.10 (5/94)		
3.8	Is the calibration curve prepared from a blank and three standards and verified with the highest standard to be within ± 5% of the actual value?	(SW846)6010A,7.3 (7/92)		
3.9	Is the upper limit of the linear dynamic range for each wavelength determined from a linear calibration prepared using the normal analytical operating procedures and verified annually by analyzing succeedingly higher standard concentrations until the observed concentration is no more than 10% below the stated concentration?	(ORDM)200.7,9.2.2 (5/94)		
3.10	Are all measurements made within the linear dynamic range where spectral interference correction factors are valid?	(SW846)6010A,4.2 (7/92)		
3.11	Is the rinse blank used to flush the instrument uptake system and nebulizer between standards, check solutions and samples to reduce memory effects?	(ORDM)200.7,7.10 (5/94)		
3.12	Is the system flushed with a rinse blank for a minimum of 60 seconds between each standard and are replicate measurements made of the blank and high standard to prove an optimal distribution of the calibration standards?	(ORDM)200.7,11.4.4 (5/94)		
3.13	Is the plasma solution used to determine the optimum viewing height of the plasma above the work coil for vertically configured plasmas?	(ORDM)200.7,10.2 (5/94)		
3.14	Are the interelement spectral correction factors verified annually using spectral interference check solutions?	(ORDM)200.7,7.13 (5/94)		
3.15	Is the nebulizer gas flow the same (less than 2% change) from day to day?	(ORDM)200.7,10.2.6 (5/94)		
3.16	Is aqueous total recoverable sample preparation performed on a hot plate at 85°C for about 2 hours (when using a 100 mL aliquot of sample) followed by covering with a watch glass and a 30 minute gentle reflux ?	(ORDM)200.7,11.2.4 (5/94)		
3.17	Is the hot plate temperature control adjusted so that an uncovered beaker containing 50 mL of water in the center maintains a temperature of no higher than 85°C?	(ORDM)200.7,11.2.3 (5/94)		
3.18	Is the calculation of dry weight based on percent solids dried at 60°C to a constant weight to avoid the loss of volatile metallic compounds or does the data user, program or laboratory report percent solids by drying to 105°C?	(ORDM)200.7,12.6 (5/94)		

3.19	Is the sample size for solid sample preparation adjusted based on the moisture content of the solid and is the final data reported to indicate wet weight or dry weight concentration at the temperature measured?	(ORDM)200.7,11.3.1 (5/94)		

Item	Section 4 - Sample Handling Practices	Reference	Yes-No or NA	
4.1	Is the pH of all aqueous samples checked to be less than 2 prior to aliquoting for processing or direct analysis to ensure the sample has been properly preserved?	(ORDM)200.7,8.1 (5/94)		
4.2	Are samples for dissolved elements filtered in the field using glass or plastic filtering apparatus and the filtrate preserved with (1+1) nitric acid to a pH < 2?	(ORDM)200.7,8.2 (5/94)		
4.3	Are samples preserved with (1+1) nitric acid to pH < 2 at the time of collection or shipped to the laboratory and preserved within two weeks of collection and analysis started only after being held preserved for 16 hours?	(ORDM)200.7,8.3 (5/94)		
4.4	Are solid samples preserved by storing at 4°C with no holding time limitation?	(ORDM)200.7,8.4 (5/94)		
4.5	Are plastic or glass sample containers washed using this sequence: detergent, tap water, 1:1 nitric acid, tap water, 1:1 hydrochloric acid,tap water and reagent water?	(SW846)CH3,3.1.3 (9/94)		
4.6	Are aqueous samples for total and dissolved metals acidified to pH< 2 with nitric acid?	(SW846)CH3,3.1.3 (9/94)		

Item	Section 5 - Quality Control Practices	Reference	Yes-No or NA	
5.1	Is the laboratory reagent blank analyzed with each batch of 20 or fewer samples and found to be less than 2.2 times the analyte's MDL or less than 10% of the analyte level determined for a sample?	(ORDM)200.7,9.3.1 (5/94)		
5.2	Is an instrument performance check solution analyzed at the beginning, end and every 10 samples? Is it found to be within 5% of calibration at the start and within 10% of calibration during subsequent analysis?	(ORDM)200.7,9.3.4 (5/94)		
5.3	Is the calibration blank less than the analyte instrument detection limit (IDL) but greater than the lower 3-sigma control limit of the calibration blank?	(ORDM)200.7,9.3.4 (5/94)		
5.4	Is the calibration blank prepared by acidifying with the same concentration of acids found in the standards and samples, analyzed after every 10 samples, and found to agree within 3 standard deviations of the mean blank value?	(SW846)6010A,7.5 (7/92)		
5.5	Is a quality control sample obtained from an outside source, analyzed at least quarterly and found to be within ± 10% of the stated values?	(ORDM)200.7,9.2.3 (5/94)		

5.6	Is a calibration check standard (made from a reference material or other independent standard material at or near the mid range) analyzed at the beginning and after every 10 samples and found to be within ± 10% of the true value?	(SW846)6010A,8.6 (7/92)		
5.7	Are the method detection limits determined annually or when a new operator begins work? Are they determined using 7 replicates at 2 to 3 times the estimated instrument detection limit?	(ORDM)200.7,9.2.4 (5/94)		
5.8	Is a laboratory fortified blank analyzed with each batch and is the recovery within ±15% of the analyte added to fortify the solution?	(ORDM)200.7,9.3.2 5/94)		
5.9	Is a laboratory fortified matrix performed for a minimum of 10% of the routine samples and is the recovery ± 30% or a 3 sigma recovery range calculated from the regression equations given in the method?	(ORDM)200.7,9.4.2 (5/94)		
5.10	Is one duplicate sample for every matrix in a batch analyzed for every 20 samples?	(SW846)6010A,8.4 (7/92)		
5.11	Is at least one typical sample per analytical batch selected for serial dilution to determine if interferences are present?	(SW846)6010A,8.5.1 (7/92)		
5.12	Is the dilution test used when the analtye concentration is at least 10 times greater than the instrument detection limit and within ±10% of the original determination?	(SW846)6010A,8.5.1 (7/92)		
5.13	Is a post digestion spike addition recovered to within 75 to 125% of the known value?	(SW846)6010A,8.5.2 (7/92)		
5.14	Are spiked replicate samples performed at a frequency of 5% or per analytical batch with a recovery of ± 20% of the actual value and a relative percent difference of ±20% for values greater than 10 times the IDL?	(SW846)6010A,8.6.3 (7/92)		